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NATURAL KAPOK FIBRES AND ACETYLATED JUTE/COIR FIBRES STRUCTURED BLEND SORBENT FOR OIL SPILL CLEAN-UP

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ABSTRACT

A novel structured sorbent blend pillow was developed using acetylated jute and coir fibres blended with natural kapok fibres. The acetylation of the jute and coir portion was carried out with acetic anhydride using N-Bromosuccinimide as catalyst under mild condition in a solvent free system. The results shows that the acetylation increases the oil sorption capacity of the blend sorbent to 1.5 times higher with a cleaning efficiency of about 90% when compared with synthetic sorbent polypropylene fibre as a control sample. On the other hand, the total replacement of polypropylene portion of the blend with natural unmodified kapok fibres increases the oil sorption capacities of the blend 10 times higher in the first cycle with cleaning efficiency of 95%. Therefore, jute, coir, and kapok fibres that are abundant as wastes are good for use as oil sorbent-active materials regardless of whether they are modified or unmodified to enhance their oleopholicity. Hence, can be used to substitute non-biodegradable materials in oil spill clean-up exercise.

Keywords: crude oil spill, clean-up, coir fibre, jute fibre, kapok fibre.

Introduction

Water is indispensable for all known living things, and because it serves as universal solvent, its usefulness and adaptableness assist in the performance of some important chemical reactions (Sargen, 2019) (see Fig. 1). It is well known that 70% of Earth is covered by water, only 3% of it is freshwater, of which 83% is found in frozen glaciers or otherwise un-available for use (World Wildlife Fund, 2017; USBR, 2020). Humans require freshwater the most, to drink, to breathe, for food processing, and to irrigate farm fields (World Wildlife Fund, 2017) (Fig. 2).

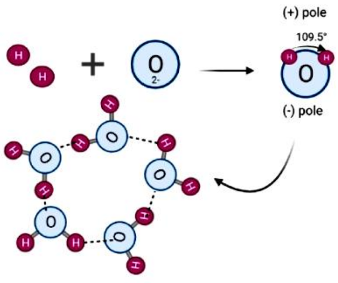

Fig. 1: Water Chemistry.

The asymmetry in the molecular structure allows water to form strong bonds with other polar molecules. It is designated as the universal solvent because of its capability to dissolve a variety of molecules (Sargen, 2019).

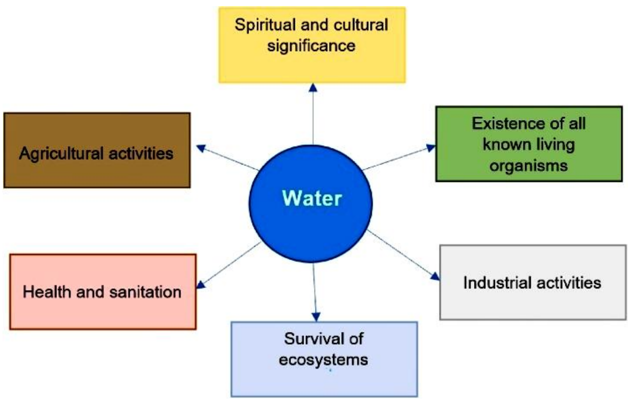

Fig. 2: Use of Water. The importance of water and applications (Ray and Iroegbu, 2021).

An oil spill is the release of a liquid petroleum hydrocarbon into the environment, especially the marine ecosystem, due to human activity, and is a form of pollution. The term is usually given to marine oil spills, where oil is released into the ocean or coastal waters, but spills may also occur on land. Oil spills may be due to releases of crude oil from tankers, offshore platforms, drilling rigs and wells, as well as spills of refined petroleum products (such as gasoline, diesel) and their by-products, heavier fuels used by large ships such as bunker fuel, or the spill of any oily refuse or waste oil (Wikipedia, 2021). Plate 1 depict the faith of oil spilled at sea showing the main weathering process.

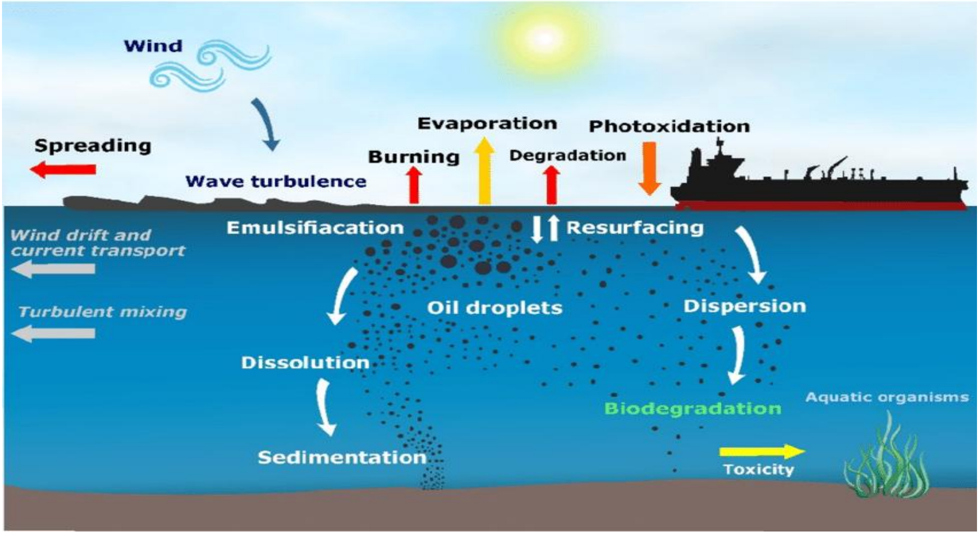

Plate 1: Faith of oil spilled at sea showing the main weathering process

Many techniques have been devised to combat crude oil spill problem. The most widely accepted by many researchers and industries is the one prepared from polypropylene fibres and is now being considered hazardous, Chung (2012). Most recent research discovery shows that natural materials are the best for oil cleaning-up (Singh et. al., 2014). Moreover, cotton and kapok are the best amongst the greens. This is because of their higher oil sorption capacity, biodegradability and recyclability. Hence, they are preeminent materials for oil spill clean-up (Singh et. al., 2014). The problem with cotton necessitated the use of modified natural fibres to enhance their oleophilicity as reported by Teli and Valia (2016) that; modified coir and jute fibres are of low cost and high-capacity effective oil sorbents. Therefore, this research investigates the use of modified jute and coir fibres with natural kapok fibres structured blend sorbent material for crude oil spill clean-up.

Materials and Methods

All chemicals are analytical grade and were used as purchased. Acetic anhydride and N-Bromo Succinimide (NBS) were purchased from Zayo Sigma Aldrich, Paderborn-Elsen, Germany. Crude oil sample and Sea water from offshore and the creeks were obtained from Research Laboratory of Nigeria Petroleum Development Company (NPDC) a subsidiary of Nigeria National Petroleum Company (NNPC), Warri Delta State, Nigeria. Extracted Coir fibres was purchased from Railway Market, Kaduna, Kaduna State, Nigeria. Jute Fabric was purchased from Ghana Cocoa Board, Jute factory Ghana. Kapok fibres were purchased from “SarkinRimi” of Zazzau Emirate, Zaria, Nigeria. Polypropylene raw fibres were obtained from NASCO Group of Companies Limited, Jos, Plateau state while Polypropylene Fabric was purchased from Abubakar Rimi Market, Kaduna, Nigeria.

Materials Preparation

Preparation of Jute fibre

The jute fabric was washed in acetone severally to remove any impurities and other additives that might have been added during finishing of the sacks and left to dry in the laboratory for 24 hrs, followed by oven drying at 65°C overnight.

Preparation of Coir fibre

Fibres recovered from fully ripened coconut were extracted from the pulp or husks surrounding the coconut called coir fibre were washed severally in fresh laboratory water and allowed to dry for 24 hrs. Coir fibre ranging from 10-23 cm were sorted and was further washed in acetone and left to dry in oven at 65°C overnight.

Preparation of Polypropylene fibre

The Polypropylene fabric was treated in the same manner as described above for jute fabric, while the polypropylene fibres of 9 cm length were used without further purification.

Acetylation of Jute and Coir fibre

Acetylation of jute and coir fibres was carried out separately in a Boros reaction flask (500ml) the flask was placed in a water bath for temperatures below 90°C, while temperatures above 90°C-130°C the reaction flask was placed in oil bath. The reaction conditions were varied with acetic anhydride and the N-Bromosuccinimide as catalyst under mild condition in a solvent free system using the method reported by Sun et.al. (2004). The temperature range was 30-130°C, catalyst concentration between 0 -1%, reaction time ranged between 0.5-6.0 hrs. After the reaction for the required time the flask was removed from the bath and the hot reagent was decanted off and thoroughly washed with ethanol and acetone in order to remove untreated acetic anhydride and acetic by products. The products were then dried in an oven for 16 hrs prior to weighing. Subsequently, the dry materials were weighed to determine the weight gains. The weight percentage gain (WPG) of the acylated Jute and coir fibre samples was calculated accordingly using equation 1:

$$\text{WPG}\ (\%) = \frac{\text{final weight} - \text{initial weight}}{\text{initial weight}} \times 100 \qquad (1)$$

Characterization

The materials were characterized using Fourier Transform Infra-red (FTIR) analysis. Comparism of FTIR Plots of modified Jute fibre and unmodified jute fibre is shown in Fig. 3 (a and b) while Fig. 4 (a and b) shows the comparism of FTIR Plots of unmodified coir and modified coir fibre respectively.

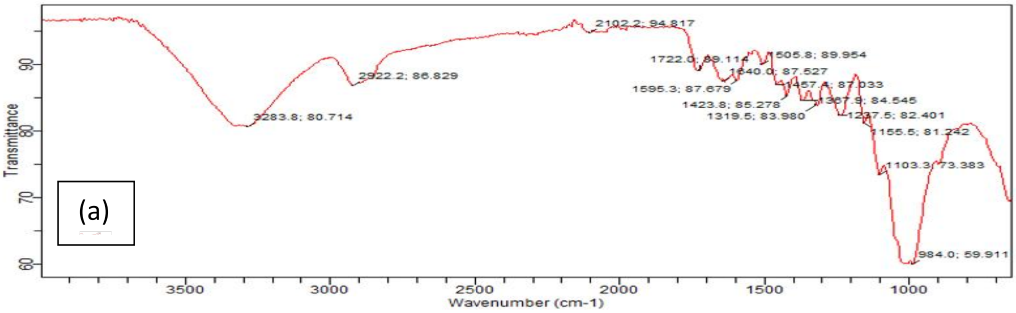

Fig.3(a) unmodified

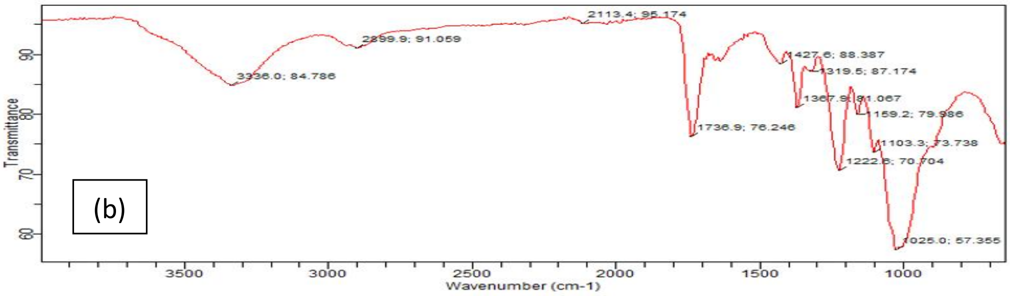

Fig. 3(b) modified Jute fibre

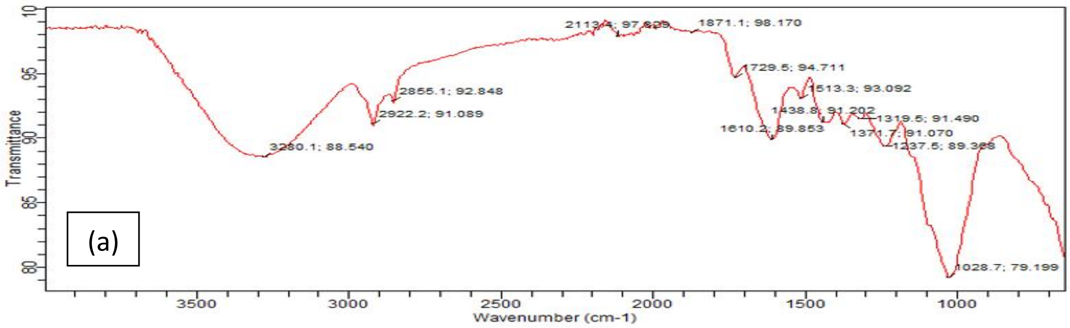

Fig. 4(a) unmodified

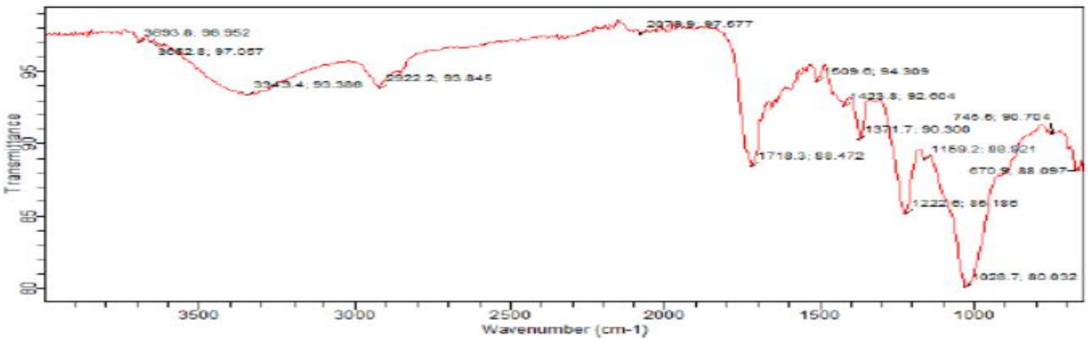

Fig. 4(b) modified coir fibre

Production of Polypropylene Fibre Pillow

Total and the French Navy standard protocols were used to fabricate the polypropylene fibre/fabric structured pillow. The housing of the pillow was 50% polypropylene fabric while the stack fibres inside the pillow constitutes 50% of polypropylene fibres. The pillow parameters were of 11cm x 11cm x 11cm x 11cm and 7mm thickness. The fabricated polypropylene fibre pillow was used as the control sample for the oil spill clean-up.

Fabrication of Structured Fibre Blend Pillow

The design was in the same manner and in conformity as the control sample, however, the backing of the pillow has now been replaced with jute fabric while the inner portion of the pillow constitutes Polypropylene fibres, Coir fibre, and Kapok fibre. Several blending ratios were fabricated i.e., Polypropylene/Jute/Coir (PP/JT/CR) Blend ratios of 45/50/5, 40/50/10, 35/50/15, 30/50/20, 25/50/25.

Buoyancy test for blend sorbent pillows

In order to determine buoyancy of the blend sorbent pillow according to the United States (US), Standard produced by American Society of Testing and Materials ASTM F 716-07, 2007, control sample and the blend sorbent pillow sample were placed on top of fresh water in laboratory as a preliminary test before being placed on the top of sea water in the glass vessel of 30 cm width and 36 cm depth separately. The buoyancy test was carried out by varying the blending proportion of the fibres blending ratios with the view to obtained optimum floatation for up to a minimum of three hours afloat without sinking below the top layer of the water level in the vessel.

Determination of water Absorbency for blend sorbent pillows

The initial weight of the fabricated blend sorbent pillow samples of different blends were recorded separately. The crude oil/sea water proportion was 1:10 as described in the Technical Manual of the American Association of Textile Chemist and Colorist (AATCC). The fabricated blend sorbent pillow samples were placed in the glass beaker tank and within each 30 minutes interval the absorbed weight was recorded. The process was continued separately for each fabricated blend sorbent pillow sample until the sample becomes saturated. Same procedure was followed for the different blend ratios under static and dynamic conditions.

Determination of optimum crude oil absorption

The crude oil absorption test was carried out firstly using the raw blending components individually, separately under static sorption condition with the blending components separately placed in 1000 ml beaker containing crude oil/sea water using 1:10 proportion respectively. The dynamic sorption condition was experimentally simulated to reflect the marine site condition. The speed of a wave in knots is 1.4 times the square root of its wavelength in feet (Nerf, 2020). Hence, Dynamic shaker was used to simulate the speed of wave onshore at the creeks. Therefore, Dynamic shaker model KS 130 NO:0002980000. was used for this study, the shaker was set at 50 mot/minute swivel motion to simulate the speed of wave. The recovery of the crude oil from the raw blending components were carried out in cycles at 30 minutes intervals until the saturation point of different samples were reached.

Results and Discussion

Properties of Blending components Fibres

The significant chemical properties in respect of the fibres and fabrics used in the fabrication of the blend sorbent pillow are shown in Table 1, 2 and 3.

Table 1: Characteristics of Zaria Kapok

S/No	ITEM	PERCENTAGE (%)
1	Cellulose (%)	63.50
2	Lignin (%)	13% of fibre mass
3	Pentosans (%)	23
4	Fibre length (mm)	27.08
5	Moisture content (%)	10.80
6	External structure	Cylindrical, very smooth
7	Density, g/cm^3	0.29

Source, Dauda and Kolawole, 2003.

Table 2: Chemical composition of coir fibres

S/No	ITEM	PERCENTAGE (%)
1	Water soluble	5.25
2	Pectin related compounds	3.00
3	Hemicellulose	0.25
4	Lignin	45.84
5	Cellulose	43.44
6	Ash	2.22

Source: Verma et. al., 2013

Table 3: Lignocellulosic content of waste jute fiber.

	Weight (%)
Pectin	2.6
Hemicellulose	20.1
Lignin	3.8
Cellulose	73.5

Source; Erdoğan U.H and Duran H., (2017).

Results in Table 4 shows polypropylene fibre length of 9cm, warp/ weft yarns of 50 tex each and 2.3 t.p.cm used to construct the fabric using Plain weave design 1/1. Coir fibre have an average length of 7.6 cm its brown and bristle grade see Plate 3.1-3.3 as also reported by (Kavitha, 2015). Jute fibre has an average length of 11 cm. Kapok fibre has an average length of 2.8cm similar results reported by (Sunmonu and Abdullahi, 1992). The jute fabric was constructed using Twill weave (2/1) with coarse yarns warp (454 tex) and weft (906 tex).

Properties of Crude oil and sea water samples

A crude oil and sea water offshore/creeks onshore samples used for this study was obtained from Nigeria Petroleum Development Company (N.P.D.C) Laboratory, Warri, Delta state, Nigeria, the three different samples of crude oils i.e., High, Medium, and Low Densities were characterized by using Rheoemeter at NPDC laboratory, Warri. The significant properties are highlighted in Table 5.

Plate 2: Polypropylene fibre.

Plate 3: Jute fabric

The obtained crude oil samples from N.P.D.C. Research Laboratory, Warri, Delta state, Nigeria were compared with the ones reported by Duong *et al.,* (2016) the nomenclatures were assigned considering two measure parameters viscosity and density which were determined by rheometer. Table 5 shows the results. Medium density, Te Giac Trang (TGT) has higher viscosity and higher hydrocarbons than high and low densities. Since, the viscosity of oil is its resistance to flow, viscosity is dependent on oil spill clean-up-operations. The sea water on-shore (creeks of Atlantic Ocean) shows higher contamination with hydrocarbons as indicated by several peaks [3309 cm^{-1} OH, 2963 cm^{-1} CH_3, 1725cm^{-1} -C=O, 1274 cm^{-1} C-O, 1636 cm^{-1} C=O, 1379 cm^{-1} C-O, 1121 cm^{-1} C-O and 1073 cm^{-1} C-O].

Single Absorption region of 1736.9 cm^{-1} was observed for the FTIR of modified jute fibre indicating C=O stretch. Hence, jute fibre modification has occurred. while the decrease in the intensity of the broad band at about 3283.8 cm^{-1} to 3336.0 cm^{-1} is assigned to cellulose O-H vibration showing decrease in water absorption. Absorption range at 1718.3 cm^{-1} for FTIR of the modified coir fibre indicates modification has occurred with decrease of intensity from 3280.1cm^{-1} to 3343.4 cm^{-1} of the O-H group assigned to cellulose decreasing significantly. Hence, enhancement of the character of the coir fibre.

Table 4: Physical Properties of Fibres and Fabrics.

Property	Fibre			
	Polypropylene	**Coir**	**Jute**	**Kapok**
Average fibre length (cm)	9	7.5	11	2.8
		Grade: (Brown & Bristle)		
Count of warp yarn	50 tex		454 tex	
Count weft yarn	50 tex		906 tex	
Twist direction of warp yarn/t.p.cm	Z / 2.3		Z / 2.1	
Twist direction of weft yarn/t.p.cm	Z / 2.2		Z / 1.2	
Type of weave	Plain- 1/1		Twill-2/1	

Table 5: Specifications of the Crude Oil

Sample of Crude oil	K.Viscosity (m^2/s)	Speed (m/s)	Torgue (NM)	Temp. (0C)	Density (g/cm^3)
High: Ruby (R.B)	1.33	30	0.1	24.5	0.8965
	0.67	60	0	24.5	
Medium:	1.45	30	0.3	24.2	0.65
Te Giac Trang (TGT)	0.777	60	0.1	24.1	
Low: Rang Dong (RD)	1.33	30	0.08	24.4	0.588
	0.67	60	0	24.5	

Buoyancy, water and crude oil absorption

ASTM F 716-07, 2007 was used to determine the performance of the blend sorbent pillow on sea water. Bullas *et al*, (2014) reported that; the test method gives high sensitivity to moisture. Hence, the test was prepared for this research. Buoyancy or Upthrust is an upward force exerted by a fluid that opposes the weight of an immersed object as describe by Archimedes Principle. Neutral Buoyancy occurs when the blend sorbent pillow average density is equal to the density of the sea water in which it was immersed, resulting in the buoyant force balancing the force of gravity that would otherwise cause the blend sorbent pillow to sink. The blend pillow sinks if its density is greater than the density of sea water in which it is immersed or rise if its density is less. The blend pillow sorbent that has neutral buoyancy will neither sink no rise but stays afloat on top of sea water as observed during the tests.

Results in Table 6 and Figure 6 shows the buoyancy decreases with increases of polypropylene fibre replacement because of the hydrophobic properties of polypropylene fibres.

Table 6: Buoyancy for the control and blend samples sinking depth and water absorption.

S/No.	Blending ratio PP/CR/JT	Period (hours)	Sinking Levels (mm)	Water absorption (owf) %
1	100	4200	0.3	0.01
2	45/5/50	4200	7.9	38
3	40/10/50	168	16.0	76
4	35/15/50	24	23.0	114
5	30/20/50	7	31.9	152
6	25/25/50	3	39.9	191

The AATCC, 79-2000, describes absorbency as an important factor that determines the suitability of a fabric for a particular use and is defined as the propensity of a material to take in and retain a liquid, usually water, in the pores and interstices of the material. Normally, the property of fabric absorbency is important when the fabric is used for special functional garments or products. The test was focused on the effect of time with the saturation point.

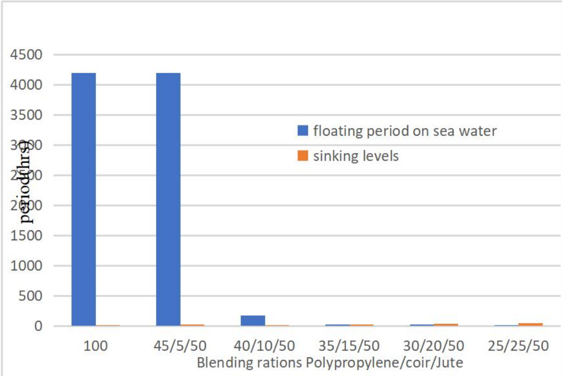

Figure 6: Buoyancy test of control sample and blend pillow samples

The results of the water absorption of 0.01% of the weight of the fibres/fabric (o.w.f %) in respect of the polypropylene fibre control sample, the water absorption increases as the percentage replacement of the polypropylene fibres was increase by 5% each of coir fibre up to 25% total replacement.

The result shows 191% water absorption for the Blend ratio (PP25%:CR25%:/JT50%), there was increase in water absorption as the period of contact with the water increases, the findings was as a result of the replacement of the polypropylene fibres by hydrophilic natural fibres i.e.; coir and jute fibres. Similarly, the sinking level below the surface of the top water level increases from 0.3mm (control sample) up to 39.9mm for the blend ratio stated above. However, the blend sample buoyancy was sustained for 3hrs at this level.

Figure 7 results presents the crude oil recovered during five cycles and the reusability of raw samples, polypropylene shows low range of recovery per cycle i.e.; 12.3g/g -15.9 it can be used for 5 cycles without decrease in recovery power.

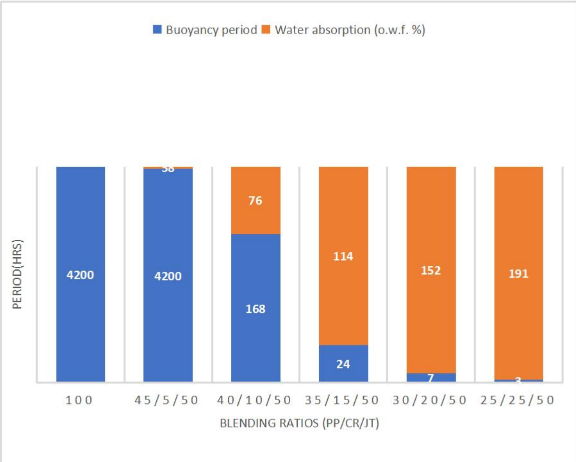

Figure 7: Buoyancy/water absorption test of control sample and different blend pillows.

Kapok fibre shows highest recovery with highest range of recovery per cycle i.e.; 31g/g – 21.4g/g, however, the recovery power falls suddenly after the 3rd cycle as result of shrinkage after every cycle. The high buoyancy of kapok is also as a result of the wax content which influences buoyancy and hydrophobicity couple with the presence of long/branched hydrocarbon chains in the kapok fibre structure (Srinivasan *et. al*.2008., Khan *et. al.*, 2004., Likon *et. al.,* 2013). The result for coir fibre recovery range was 1.1g/g – 2.6g/g showing low recovery power and low range, similarly, jute fabric exhibits lowest range of recovery of 0.9g/g - 1.1g/g the recovery per cycle was lowest for jute fabric as a result of its hydrophobic property and perhaps the jute fabric used was pretreated with repellent agent for storage purposes.

Figure 8 shows the results of polypropylene control with recovery under static conditions up to 15.715g/g while the Blend ratio (PP25%:CR25%:/JT50%) has 3.686g/g highest recovery in the first cycle.

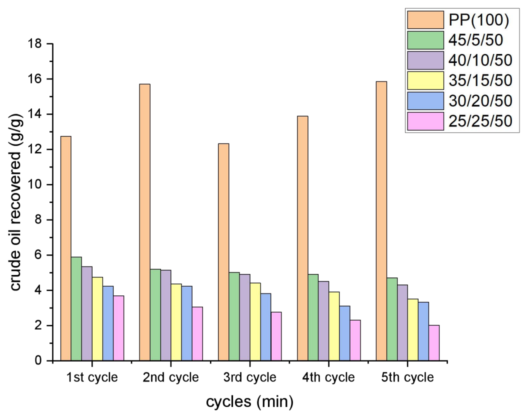

Figure 8: Crude oil absorption capacity and reusability of blend sorbent pillows (static condition)

The overall recovered crude oil per cycle decreases as the recycling continues for all blend sorbent pillows indicating best recoveries from 1st – 3rd cycles, except polypropylene control sample which can be reuse for up to 5th cycle. However, under static conditions the recovery of crude oil was higher as a result of agitation of the blend sorbent pillows indicating up to 15.93g/g for polypropylene control sample while Blend ratio (PP25%:CR25%:/JT50%) has 5.8g/g it was observed under dynamic conditions there was no wide range of recovery per cycle. The cleaning efficiency of the polypropylene control sample and Blend ratio (PP25%:CR25%:/JT50%) sample under static and dynamic conditions were 90%,95% and 39%,30% respectively, the lower cleaning efficiency from the recovered crude oil for the Blend sample ratio (PP25%:CR25%:/JT50%) was as a result of the hydrophilic nature of the blend sample and the retention of the crude oil adsorbed by the natural fibres which are more difficult to be released from the fibres/fabrics, while polypropylene fibre releases crude oil adsorbed much easier when compressed.

Finally, Plate 2 showed fabricated structured jute/coir/polypropylene fibres and fabric blend sorbent pillow.

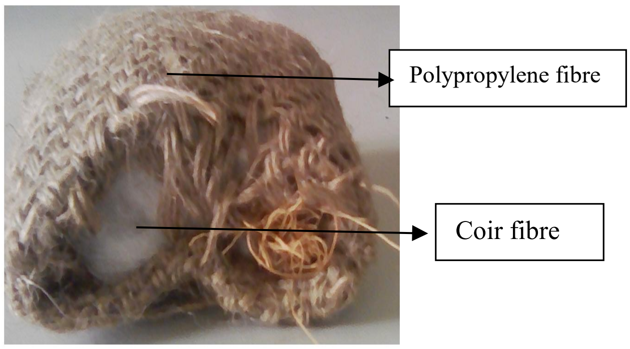

Plate 2: Fabricated structured jute/coir/polypropylene fibres and fabric blend sorbent pillow.

Conclusion

Jute and coir fibres has been modified successfully with acetic anhydride to replace the hydroxyl groups. Hence, induced hydrophobicity to the fibre surface.50% jute fibres and 25% coir fibres modified as a replacement of polypropylene fibres portion of the blend pillow has been achieved, which resulted to 47%-50% cleaning efficiency under static and dynamic conditions.

Advantage of biodegrability of new blend pillow by replacement of 75% synthetic polypropylene fibres portion with natural modified fibres for crude oil spill clean-up exercise. Hence, a new blend pillow has been fabricated that can be recycle three (3) times for use at creeks, waterways, rivers, jetties, beaches, lagoon, waterfronts.

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